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Notes on Setora nitens Walk., a "Nettle Caterpillar" Pest of Coconut Palms, with Special Reference to Outbreaks in the Teluk Anson and Bagan Datoh Districts.

BY

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Introduction.

Setora nitens (Lepidoptera, Heterocera, Limacodidae) was first described by Walker in 1855 (1).

The first occasion on which specimens were obtained by the Entomological Division of this Department was July, 1923. They were then despatched to the Imperial Bureau of Entomology London, and specific determination was received in September of that year. Since then only brief mention of this species has been made from time to time (2—9).

From information received from planters, it would appear that this pest has been known in the Teluk Anson and Bagan Datoh Districts (Lower Perak) since 1912, but, owing to the short duration of the outbreaks and to the slight damage caused by it, it has not been regarded in the light of a serious pest.

From March, 1928, attacks of this pest have been more frequent and more widespread, and it has extended its range in some places, from young palms to mature palms. Finally, the situation has been such as to give rise to considerable alarm on the part of some of the planters in those districts.

Of the whole area, there are approximately two thousand five hundred acres in which the pest is or has been present, in varying degrees of abundance.

The writer is greatly indebted to Mr. F. R. Mason, Agricultural Field Officer, Perak South, for much information regarding the acreage and infected areas of the estates which he visited in March 1929, with Mr. Mason, for the purpose of obtaining more details regarding the life history of this insect, and also to explain control methods to the planters concerned.

The control methods recommended were generally adopted with enthusiasm, and it is believed that the results obtained will fully justify all additional expenditure of time and money.

It must be pointed out that it would be foolish to be unduly optimistic in expecting immediate results from the control methods recommended, for when once a pest has obtained such a firm hold, as has the pest in question, reduction in its numbers, or its final elimination, (which is doubtful), is likely to be spread over a considerable period.

Ultimate success depends very largely on the co-operation of all persons concerned in the affected areas.

This pest at present seems to be confined almost entirely to the plantations north of the Teluk Anson—Bagan Datoh road. There are slight traces of infestation on palms near the roadside in the Malay Reservation south of the road, but the damage done is negligible.

The spread of the larvae from young palms, i.e. palms up to about six years of age, appears to be of comparatively recent date. It is highly probable that the spread has taken place, to some extent, from "supplies" planted in the mature areas.

Distribution and Host Plants.

So far, *S. nitens* has been recorded in this country, in addition to the Teluk Anson District, from Kuala Kurau and Sabak Bernam on coconut palms, from Serdang and the districts referred to in the present paper, on oil palms (*Elaeis*

guineensis Jacq.), and from the Kuala Selangor district on Nipa (*Nipa fruticans* Wurmb.)

In the jungle areas adjacent to coconut plantations in the Bagan Datoh district there are large numbers of nibong palms (*Oncosperma tigillaria Hort.*), and it was suspected that they might be another alternative food plant of *S. nitens*.

A very careful search failed to yield any larvae of this species, although one larva of an allied species was found.

In Java and Sumatra, *S. nitens* has a far greater range of foodplants, for, in addition to coconut, oil and nipa palms, it has been recorded on Cacao (*Theobroma cacao* Linn.), tea (*Camellia thea* Link.), Coffee (*Coffea* spp.), Chinchona (*Chinchona Ledgeriana* Moens.), Tobacco (*Nicotiana* spp.), Banana (*Musa* spp.), Pomelo (*Citrus Decumana* Murr.), Rambutan (*Nephelium lappaceum* Linn.), (10).

The Ovum.

The ova, (Plate 1 fig. 1) which are elliptical and very strongly compressed in shape, and shining yellow when first laid, are deposited on the under surface of a pinna, and usually near its tip. The surface of the shell is very finely reticulate.

The moth sometimes deposits her ova in small irregular batches, and, when laid in this manner, they slightly overlap each other.

Owing to their shining and semi-transparent appearance they are most difficult to discover. It is thought highly probable, that the empty egg shell provides the young larva with its first meal (as is frequently the case with other lepidopterous insects) for, even on fronds on which numerous young larvae have been found, no egg remains have been detected. Should the empty shell not be consumed by the young larva, it shrivels up and in that state is almost unrecognisable.

Occasionally, after a prolonged and careful search, remains of the adhesive substance, by which ova are attached to the leaf surface, have been discovered.

An ovum measures 3 mm. x 2 mm.

The Larva.

The larvae, (Plate 1 figs. 2, 3) to which the name of "nettle caterpillars" has been given, owing to their spines causing a sharp stinging sensation, analogous to that produced by "stinging nettles," if they come into contact with one's bare flesh, are readily recognised.

In colour, they are greenish yellow, or orange, or pale bluish green, with a narrow purplish stripe spotted with blue, black and yellow along the middle of the back, and with a row of purplish elongate spots bordered with yellow along the sides. These stripes are not very distinct in larvae which have only recently emerged from the ovum.

They are provided with twelve moderately long tubercles, four being placed on the front part of the body, four on the hind part and two on each side of the back. Along the lower margins of the sides is a row of shorter tubercles. All these protuberances are armed with spines capable of "stinging".

The spines are shaded with brownish black, in the young larva. In older larvae they are purplish brown.

The urticating power of the spines increases in direct proportion to the age of the larva. In very young larvae, it has been found by experiment, that the urticating sensation is barely perceptible. The spines of a full grown larva removed from a recently spun cocoon, however, will produce intense irritation.

On hatching, the larva measures about 2 mm. in length, and when full grown, about 35 mm.

In shape, and by their gliding mode of progression, these larvae somewhat resemble slugs. They move very slowly, taking a minute at least to cover a distance of three inches.

The undersurface of the palm pinnae is always preferred by the larvae in all stages of development. Also, they are most commonly found on the lower fronds. This preference is, no doubt, due to their dislike of direct exposure to the sun's rays, and, at the same time, in that position, they would receive ample protection from rain.

The epidermis of the pinna is that part which is first attacked. Later on, the whole pinna is devoured, leaving the midrib only. A badly attacked coconut plantation has the appearance of having been partially destroyed by fire.

On reaching maturity, the larvae often fall off the frond to the ground, and shortly afterwards seek out suitable spots for the spinning of their cocoons.

The Cocoon.

The cocoon, (Plate 1 figs. 4, 5) which is completed by the larva in a little more than half an hour, is spherical in shape and light brown in colour. The diameter varies from 8mm. to 12 mm. Its surface is smooth, rather irregular, and has a dull gloss.

Cocoons have been likened by some people to small new potatoes. They resemble more closely, however, the round galls known as "oak apples" which are found on oak trees in Europe.

Various situations are chosen by the larvae when pupating. Cocoons are constructed in cracks in the soil, among roots of cover crops, among the exposed roots of the palms and among the fibre at the base of the fronds. Frequently groups of eight to a dozen cocoons have been found in a mass among the fibre. On several occasions, the writer came across a cocoon spun up in the empty cocoon of a larva of a previous generation.

The Pupa.

The colour of the pupa, (Plate 1 fig. 6) immediately after transformation is pale yellowish white. Shortly afterwards, the wing coverings, the legs and the thorax become brown, or greyish brown, but the abdomen retains its pale yellowish white colour. Prior to the emergence of the moth, the whole pupa becomes dark greyish brown. The eyes, wings and legs are distinctly visible, the legs being free, i.e. it is possible to lift them away from the body without previously cutting the integument of the pupa.

The surface of the pupa is smooth and has a greasy appearance.

Pupae vary in size according to the sex of the moth, those of the males being the smaller. They measure 9—11 mm. to 15—17 mm. in length.

The Moth.

The moth, (Plate 1 fig. 7) is a somewhat robust insect, having a wing expanse of 30—35 mm. The forewings are brownish and somewhat glossy and they have a broad, somewhat indefinite dark brown transverse band near the apex and a narrow brown diagonal stripe. The hind wings are of a unicolorous paler brown.

Life Cycle.

The life cycle of *S. nitens* has not been completely worked out, but it is thought that it occupies a period of 6—7 weeks. Twenty five to twenty seven days is the duration of the pupal stage.

In captivity the larvae do not thrive well, and frequently, even when it has been possible to keep them alive until they have spun cocoons, many of them dry up or decompose before pupating.

Isolating them in individual cages may yield better results, for, it is probable, that the larvae are able to inflict injuries on each other by contact with each other's spines.

From observations made in the field, it would appear that there is some overlapping of broods, since larvae in all stages of development have been seen at the same time.

It is important, therefore, that frequent inspections of all areas be carried out.

The life cycle of the parasite referred to is of approximately the same number of days as that of *S. nitens*.

Mechanical Control.

Handpicking the larvae and cocoons is considered the most efficacious means of ridding a plantation of this pest, when it is confined to palms up to about six years of age.

When dealing with very young larvae, it will be found that squashing them *in situ* will be more practicable than picking them. It must, of course, be borne in mind that the hands of persons employed on this work must be adequately protected, from the poisonous spines of the larvae, by binding with strips of rag.

Larger larvae may be dislodged from the leaves with a stick or a pair of tweezers, and, when the tins, with which the collectors should be provided, are full, the contents should be destroyed by burying in the soil.

The problem of control presents greater difficulties when the attack has spread to mature palms. The employment of children armed with long sticks to knock off the larvae, has been tried, but, this method is cumbersome and liable to inflict much damage to the leaves.

As an improvement on this, it is recommended that long bamboo poles, to which a rough brush is fixed, be used to brush off larvae, which, undoubtedly, would be injured in the process, and would be unable to regain the palms from which they had fallen.

The brush part of this implement could be constructed from material at hand, such as coconut husk fibre.

The systematic collection of cocoons is of immense value in reducing the numbers of the pest, and can be allotted with advantage to women and children. Where there are dense cover crops however, the task is more difficult, as suitable places under which the larvae pupate, are provided.

Collected cocoons should not be destroyed. The reason for this is set forth under the section "Biological Control".

Spraying with a lead arsenate solution, mixed in the proportions of 2 to 3 lbs. to 50 gallons of water has proved effective, but it is not favoured as a control measure, owing to cost, the length of time taken to spray a comparatively small area, and to the necessity of expert supervision.

Again, spraying can be really efficacious only when one is dealing with young palms, unless a very powerful type of sprayer be used, but then, the difficulties in negotiating

the crossing of drains with a machine of this type would be insurmountable.

Biological Control.

The larvae of *S. nitens* are attacked by several parasites, one of which, a large grey and black fly, known as *Chaetexorista javana* B.B., (Plate 1 figs. 8, 9) is not unlike a "blow fly".

Chaetexorista javana, (Diptera, Tachinidae) was first described by Brauer and Bergenstamm (Denkschr K. Akad. Wiss Wien. Lxi p. 616) in 1894 and a further description of the adult, as well as a description of the puparium was made in 1925 by Bezzi (11), from material submitted by the Entomological Division of this Department.

This fly, if encouraged, will, no doubt, prove a valuable ally in combating the pest. As a check on *S. nitens* feeding on tea in Java, it has proved itself efficient (12).

This fly deposits eggs on all stages of the larvae of *S. nitens*. The maggot on hatching bores through the skin of its host and feeds on the nutritive fluids of the body. Finally the *S. nitens* larvae succumbs, but not as a rule before spinning its cocoon.

The maggot of the fly then pupates, and, in the space of two to three weeks the adult fly emerges by pushing off the cap at one end of the cocoon, in the same manner as the moth would do.

As previously mentioned, therefore, collected cocoons should not be destroyed, but should be placed in a single layer in trays covered with wire gauze having a mesh of about 5 mm. The moths emerging from non-parasitised cocoons will be prevented from escaping from the trays owing to the smallness of the mesh of the gauze, whereas the parasitic flies will have no difficulty in doing so, and thus will be free to carry on the work of destroying other larvae. As a substitute for gauze which may be hard to obtain, with a 5 mm. mesh, sheets of tin suitably bored will be satisfactory for covering the trays.

Trays which are utilised for this purpose should be supported on trestles, the legs of which are placed in tins contain-

ing water to which is added some kerosene or disinfectant. By this means, the trays are isolated from the attacks of ants, which, while destroying moths, will also destroy freshly emerged parasites, and by so doing will defeat the object of the scheme.

It is also necessary to protect the trays from sun and rain by constructing a shelter with a corrugated iron, or palm leaf roof.

Up to the present, no parasite of the ovum has been discovered.

Insectivorous birds, or birds which are more or less omnivorous such as crows and mynahs should be encouraged (13). One species of mynah (*Acridotheres javanicus* Cab.) is reported from Java as feeding on *S. nitens* pupae (14) possibly therefore, the local species *Aethiopsar fuscus torquatus* Rob, and Kloss (15), may be reckoned as a bird to be encouraged.

In the field, rats destroy a large number of cocoons, and they have been disturbed in the act of removing cocoons from trays, of which the construction was slightly defective. Ants also will penetrate freshly constructed cocoons and destroy the occupants.

Fungoid diseases appear to be responsible for the destruction of large numbers of larvae, but more information regarding them is required before they can be utilised as methods of control.

An examination of two thousand cocoons (about a tenth of the number collected on one estate) gave the following figures:—

Cocoons from which moths had emerged.	Cocoons containing dead larvae.	Cocoons containing dead pupae.	Parasitized.
1114.	543.	141.	202.

A smaller number of cocoons collected on another estate gave the same percentage of parasitization.

The larvae found dead were invariably covered with fungoid growths, the pupae only occasionally so, and the cocoon itself was usually damaged, having apparently been eaten through by ants before it had hardened.

Parasitization, as will be seen from the figures given above, is responsible at present for a comparatively small percentage of deaths, but, the writer is confident that a far greater percentage will be obtained, if the scheme of parasite breeding and liberation is carried on without intermission. In Java, parasitization in one instance reached the high figure of 88% (10).

Summary.

Notes on and descriptions of the early stages and of the adult of *S. nitens* Walk. and of a parasite *Chaetexorista javana* B.B. are given.

This "nettle caterpillar" pest of coconut palms has increased in numbers in recent years in the Bagan Datoh district.

It has also extended its range in some places from young palms to mature palms.

This spread is thought to take place partly from "supplies" planted in mature areas.

The Coconut plantations in the Malay Reservation on the South side of the Teluk Anson—Bagan Datoh road are only very slightly attacked.

Systematic collection of larvae and cocoons, and the breeding and liberation of parasites are considered the most efficacious methods of control.

Conclusion.

The writer wishes to express his thanks to all Managers and Assistant Managers of the Estates on which these observations were made, for their help and hospitality.

References.

1. Eastern Lepidoptera Heterocera Swinhoe.
2. Annual Report 1924. Malayan Agricultural Journal, Vol. XIII, p. 223.
3. Insects on Oil palms. Malayan Agricultural Journal, Vol. XIII, p. 252.

4. Report of Chief Field Officer. Malayan Agricultural Journal, Vol. XIV, p. 152.

5. Miscellaneous Insects of 1925. Malayan Agricultural Journal, Vol. XIV, p. 251 Corbett and Gater.

6. Annual Report 1926. Malayan Agricultural Journal, Vol. XV, p. 169.

7. Oil Palm in Malaya. Malayan Agricultural Journal, Vol. XV, p. 341.

8. Miscellaneous Insects in 1926. Malayan Agricultural Journal, Vol. XV, p. 418 Corbett and Dover.

9. Annual Report 1927. Malayan Agricultural Journal, Vol. XVI. p. 136.

10. Parasites and Predators in Malaya. Malayan Agricultural Journal, Vol. XVI, p. 420 Corbett and Miller.

11. Agricultural Zoology of the Malay Archipelago. K. W. Dammerman.

12. Some Tachinidae (Dipt.) from the Federated Malay States, Bulletin of Entomological Research, Vol. XVI, 1925. M. Bezzi.

13. Parasieten van Thee en Kina insecten (Parasites of Insects on Tea and Cinchona). De Thee. vi, No. 4 pp. 115—122. Buitenzorg, December 1925 R. Menzel.

14. The Food of Birds in India. Memoirs of the Department of Agriculture in India, Vol. III, 1912. C. W. Mason and H. Maxwell-Lefroy.

15. Vogels als Naturlijke vijanden van Schadelijke insecten (Birds as Natural Enemies of Injurious Insects) De Thee. III, No. 2, pp. 50—52, Buitenzorg, June 1922 R. Menzel.

16. Birds of the Malay Peninsula. H. C. Robinson.

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Explanation of Plate.

Figure 1. Ova on pinna of Coconut.

,, 2. Larva a few days old.

,, 3. Full grown larva.

,, 4. Cocoon.

,, 5. Cocoon from which moth has emerged.

,, 6. Pupa.

,, 7. *Setora nitens*, Walk. Female.

,, 8. Puparium of *Chaetexorista javana* B. and B.

,, 9. *Chaetexorista javana*. B. and B.

(All figures enlarged.)

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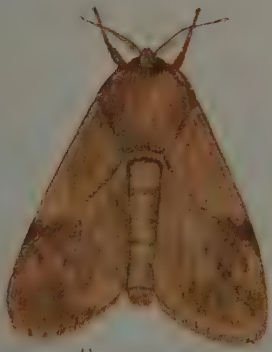

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The Periodic Harvesting of Tuba Root.
(*Derris elliptica*, Benth.)

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Introductory.

The growing use of tuba, *Derris* spp., as an insecticide has resulted in an increased interest being taken in the cultivation of this crop in Malaya.

The exports from the Federated Malay States during the past three years are as follows:—

Year.	Amount. (lbs.)	Value. $
1926	54,548	24,175
1927	79,065	39,942
1928	80,263	27,357

Tuba is also being cultivated to a considerable extent in Johore, the estimated planted area for the year 1928 being 2,000 acres.

Two species of *Derris* are commonly cultivated, namely *D. elliptica*, Benth., frequently referred to locally as "tuba puteh," and *D. malaccensis*, Prain, known as "tuba merah."

Although the differences between these two species are not very marked the following are distinctive vegetative characters. The undersides of the leaflets and petioles of *D. elliptica* are softly hairy, whereas those of *D. malaccensis* are glabrous. Further, the undersides of the former are usually greyish in colour while those of the latter are light green. The young foliage of *D. malaccensis* is bright red-brown. These colour characters are probably the origin of the Malay names of the two plants.

Since it is well-known that the toxicity of tuba root, measured by the amount of ether extract, varies according to the age of the roots an investigation on both species is being carried out jointly by the Agricultural and Chemical Divisions in order to ascertain firstly the extent of these variations and secondly the optimum age at which the roots should be harvested.

The present paper deals only with *Derris elliptica*, the plots of *D. malaccensis* not yet being ready for harvesting. It is hoped, however, to publish at an early date the results of some other experiments carried out on much smaller plots on the variations in the amount of ether extract from roots of the latter species.

Field Experiment.

Since tuba is interplanted frequently with some permanent form of cultivation it was decided for comparative purposes to carry out the experiment by planting the crop with kapok *(Ceiba pentandra)*. The total area of the block chosen was 5 acres, the soil being for the most part a dark coloured clay-loam. Only 4 acres were employed for the experiment, the remaining acre consisting of an outcrop of lateritic soil on which the kapok and tuba had failed to make good growth.

Planting.

The tuba cuttings were interplanted between the kapok trees, which were spaced 20 feet by 20 feet square planting. The kapok trees had been six months in the field when the tuba cuttings were planted during September 1926. The tuba cuttings were planted in rows 3 feet apart and spaced 3 feet apart in the rows. Five rows of tuba were planted between each row of kapok, running east and west, allowing 4 feet between the outside rows of the tuba plants and the kapok trees.

This planting arrangement gave 3,555 plants per acre compared with 4,840 if the tuba had been planted as a sole crop.

The tuba stems were cut into lengths of from 15 to 20 inches and placed in the soil in a slanting direction with

about two-thirds of the length of the stem below the surface. A percentage of the cuttings failed to take root and were supplied during favourable weather.

Further experience at the Government Plantation, Serdang, demonstrates the fact that considerably better results are obtained by rooting the cuttings in nursery beds under light shade before planting in the field. By this method not only is a larger number of rooted plants ensured, thus obviating the necessity of supplying vacancies, but the risk of failure from drought is reduced as the cuttings commence to grow within a few days of being transferred.

The block was kept clean weeded during the time of the experiment, no manure or lime being applied.

Several of the plants flowered during February/March, 1928, but as usual with *D. elliptica* none of the flowers set fruit.

Harvesting.

When the tuba root was nearing maturity, during June 1928, arrangements were made to harvest an acre at 21, 23, 25, and 27 months' growth respectively. The method of calculating this area was to take 108 squares of tuba, enclosed by four kapok trees, as an acre. Further, the method of harvesting was to divide the area into six groups of four rows, one row from each group being harvested at the conclusion of each period of growth. Since the number of kapok trees planted along the total length of six rows was 108 each set comprised one acre. Errors from variation of soil were to a great extent eliminated by means of guard rows at either end of the block and the six repetitions in each group harvested.

The first process of harvesting was to cut all stems and runners as close to the ground as possible and remove them to one side of the plot. The entire roots were then lifted, cleaned of soil and tied into bundles, which were transported to the store and weighed. The weights of each harvest are shown in Table I under the heading of "Weight of fresh roots".

During the day the roots were placed in the sun to dry and sorted into "coarse" and "fine" roots at convenient times during the process of drying.

The division of the crop into "coarse" and "fine" roots was somewhat arbitrary, the "coarse" roots being those

thicker than an ordinary sized pencil, while the remainder were considered as "fine". The reason for this division into two classes is that the "fine" roots are more acceptable on the London market, probably on account of the increased percentage of ether extract per unit weight of root. It appears that "coarse" roots are only acceptable when the "fine" roots are not offering.

Drying was continued until the roots ceased to lose weight, the details of the various harvests being also recorded in Table I. At this stage, representative samples, each weighing 25 lbs., were taken from both "coarse" and "fine" roots for analysis.

Method of Analysis.

The air-dry roots were cut into small pieces from $\frac{1}{4}$—$\frac{1}{2}$ inch long, the mass being quartered until about 3 lbs. remained. This sample was ground in a laboratory mill until it was reduced to a coarse powder, which was quartered again until approximately $\frac{3}{4}$—1 lb. remained. This quantity was ground and sieved until the whole of the material passed through a 0.5 mms. sieve. Weighed quantities of the fine powder were taken for the determination of both moisture and ether extract.

Since it is well-known that tuba root undergoes decomposition if heated for any period the moisture determination was made by drying the material over phosphorous pentoxide in vacuo to constant weight.

The amount of ether extract was determined by treating the powder with ether in a Soxhlet extractor at a temperature just sufficient to allow the ether to boil. When extraction was complete the solvent was distilled off and the residue dried to constant weight at 100°C. Although slight decomposition takes place by drying the ether extract in this manner and the results are therefore slightly low they will be comparable, since the same method was used throughout the investigation.

The results of analysis for both "coarse" and "fine" roots are recorded in Table II. In all cases the figures represent the average of duplicate determinations.

TABLE I.

Yields of Tuba Root *(Derris elliptica)* at Varying Ages.

Time of harvesting.	Age of roots. months.	Actual yield of root per acre as inter-crop.				Calculated yield of root per acre as sole crop.			
		Fresh root. lbs.	Air-dry root.			Fresh root. lbs.	Air-dry root.		
			Fine lbs.	Coarse lbs.	Total lbs.		Fine lbs.	Coarse lbs.	Total lbs.
June 1928 -	21	939	265	235	500	1277	361	320	681
August 1928 -	23	1610	452	225	677	2190	615	306	921
October 1928 -	25	1983	652	179	831	2697	888	244	1132
December 1928 -	27	2533	815	350	1165	3445	1110	476	1586

TABLE II.

Moisture Content and Ether Extract Content of Fine and Coarse Roots of Tuba Root *(Derris elliptica)* of Varying Ages.

Time of harvesting.	Age of roots. months.	Weight of air-dry roots. lbs.	Moisture content. per cent.	Calculated weight of roots on moisture-free basis. lbs.	Ether extract of roots (on moisture-free basis) per cent.	Calculated amount of ether extract. lbs.
A. Fine Roots.						
June 1928	21	265	9.49	240	7.88	18.9
August 1928	23	452	10.74	403	9.63	38.8
October 1928	25	652	11.58	576	8.59	49.5
December 1928	27	815	7.90	751	5.17	38.8
B. Coarse Roots.						
June 1928	21	235	10.29	211	6.99	14.7
August 1928	23	225	10.85	201	4.52	9.1
October 1928	25	179	16.54	149	3.18	4.7
December 1928	27	350	9.55	317	4.84	15.3

Remarks and Conclusions.

The figures in Table I indicate a gradual increase in yield in proportion to the age of the root, also that the yield of air-dry root is approximately 45 per cent. of the weight of fresh root. The moisture content of the air-dry roots is approximately 10 per cent.

The results in Table II show that for the "fine" roots the maximum figure for the amount of ether extract is obtained with roots 23 months old, the figures showing definite decreases for roots of greater age. In this connection it may be mentioned that the maximum figure for the amount of ether extract is less than that found for other samples of roots of *D. elliptica* previously examined.

The increase in the amount of ether extract for the "coarse" roots 27 months old compared with those for 25 months is probably due to a general increase in the size of the roots, resulting in a greater proportion of roots which might be classed as either "coarse" or "fine".

A further table, Table III, has been compiled from the results given in the two previous tables in order to ascertain the proportion of the weight of "fine" roots to total roots at varying ages, also the proportion which the calculated amount of ether extract for the "fine" roots bears to the total amount for the harvest.

These points are of importance from an economic standpoint, especially in view of the relatively slight decreases in the amounts of ether extract. For example, although the results in Table II show that the amount of ether extract for the "fine" roots is at its maximum after 23 months the figures in Table III show that from the point of view of agricultural practice it would be more profitable to delay harvesting until the roots are 25 months old, when both the proportions of "fine" roots to total roots and of ether extract present in "fine" roots to that in total roots are at their maxima.

Further, although the results show a definite increase in the yield of root by delaying the harvest until 27 months there is no corresponding increase in the total amount of ether extract.

TABLE III.

Proportions of Fine Roots and of Ether Extract from Fine Roots of Tuba Root *(Derris elliptica)* of Varying Ages.

Time of harvesting	Age of roots. months.	Total weight of roots on moisture-free basis. lbs.	Weight of fine roots on moisture-free basis. lbs.	Proportion of fine roots on moisture-free basis. per cent.	Total weight of ether extract. lbs.	Weight of ether extract from fine roots. lbs.	Proportion of ether extract in fine roots. per cent.
June 1928 -	21	451	240	53	33.6	18.9	56
August 1928 -	23	604	403	67	47.9	38.8	81
October 1928 -	25	725	576	79	54.2	49.5	91
December 1928 -	27	1068	751	70	54.1	38.8	72

The yield of air-dry root obtained from one acre at 25 months is found to be 831 lbs. when the tuba is planted as an inter-crop, which corresponds to 1132 lbs. per acre when planted as a sole crop. This latter figure is considered a fair average yield.

Summary.

1. The present paper deals with an investigation regarding the variations in the amounts of ether extract of tuba root, *Derris elliptica*, Benth., of varying ages.

2. The results show that taking both the yield of root and the amount of ether extract into account the optimum age for harvesting is when the plants are 25 months old.

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The Oil Content of Malayan Estate Copra.

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Introductory.

Reference was made in a previous number of this Journal, Vol. XVII, 1929, No. 5, page 127 to an investigation regarding the present oil content of Malayan estate copra.

This investigation was undertaken owing to reports received from London that the oil content of Straits copra was tending to diminish. Figures were quoted to the effect that while Straits copra formerly contained 66/67 per cent. of oil and yielded 63/64 per cent. of oil recent consignments had been found to contain 63/64 per cent. yielding only 60/61 per cent. of oil on treatment. Such a statement pointed therefore to the necessity of carrying out a series of analyses to ascertain the present average oil content of the product.

Although a large proportion of the copra exported from Malaya is produced by small cultivators it was considered advisable in the first instance to limit the investigation to the estate product on account of its greater uniformity.

Methods of Analysis.

Arrangements were made with six estates distributed along the West Coast to forward at monthly intervals for a period of six months samples of their copra as exported. These samples consisted of pieces of copra drawn from the individual sacks before stitching, each sample weighing approximately 5 lbs.

The selection of average portions of copra for analysis must be carried out carefully owing to the difference in the oil content of the white meat and the brown testa, which renders it essential to take proportionate amounts of the two constituents. In the present investigation the sampling was carried out by trimming the individual pieces of copra until approximately rectangular portions of each piece remained, when a thin slice of the material was cut transversely from one side of each piece with a sharp knife. This method, although laborious, has given good results, since it is only

occasionally that the difference in oil content of duplicate determinations exceeds 0.5 per cent.

The moisture content was determined by drying a weighed amount of the thin slices for a period of 6 hours at a temperature of 100°C. The heating was not continued since in most cases it found that after this period the material had turned brown on the edges, indicating that slight decomposition was taking place. Although therefore the figures may not represent the true moisture content of the material the results are comparable.

The dried material was treated in a Soxhlet extractor with petroleum ether. The semi-extracted copra was ground in a mortar with a small quantity of ignited sand, the mixture being replaced in the extractor for further treatment to complete the removal of the oil. The petroleum ether extract was filtered, the solvent distilled off and the oil dried to constant weight.

Moisture Content of Copra.

The following table gives a summary of the results of all the separate determinations for the samples examined from each estate. In order to make the table as concise as possible only the minimum, maximum and average figures have been given.

Estate reference letter.	No. of determinations.	Moisture content (loss at 100°C)		
		Minimum per cent.	Maximum per cent.	Average per cent.
A	12	4.79	6.45	5.68
B	9	4.88	6.59	5.77
C	13	4.68	8.75	7.24
D	10	6.13	8.43	7.52
E	11	6.52	9.08	7.56
F	7	6.89	8.44	7.60
Total	62	4.68	9.08	6.90

If the figures for all the separate determinations are divided into 1 per cent. groups, the first group having for its lower limit the whole number next below the minimum figure, 4.68 per cent. the various results can be grouped as follows :—

Group. per cent.	No. of determinations.
4- 5	7
5- 6	6
6- 7	19
7- 8	19
8- 9	10
9-10	1
Total	62

The results of analysis show therefore that the average moisture content of copra as despatched from estates is approximately 7 per cent. Further, the group table confirms this figure, since more than 60 per cent. of the determinations fall within the limits 6-8 per cent, approximately 30 per cent. of the determinations being within the group 6-7 per cent. and an equal number in the group 7-8 per cent.

Oil Content of Copra.

The following table gives a summary of the results of the oil determinations for the samples examined from each estate. In order to make the table as concise as possible only the minimum, maximum and average figures, calculated on a moisture-free basis, have been entered.

Estate reference letter.	No. of determinations.	Oil content (calculated on moisture-free basis)		
		Minimum per cent.	Maximum per cent.	Average per cent.
A	12	63.62	67.15	65.37
B	9	65.24	67.16	66.47
C	13	63.90	67.27	65.29
D	10	62.29	66.68	65.45
E	11	63.83	66.83	65.46
F	7	63.45	66.97	65.64
Total	62	62.29	67.27	65.61

If the figures for all the separate analyses are divided similarly into 1 per cent groups the results will be as follows:—

Group per cent.	No. of determinations.
62-63	1
63-64	5
64-65	14
65-66	14
66-67	25
67-68	3
Total	62

The results of analysis show that the average oil content of the samples examined, calculated on a moisture-free basis, lies between 65.5 and 66.0 per cent. This average figure is confirmed by the group results which indicate more than 63 per cent of the results of analysis to fall within the group 65-67 per cent, 22 per cent within the group 65-66 per cent and 41 per cent within the next higher group.

Relation Between Moisture and Oil Contents.

Although the figures for oil content have been calculated on a moisture-free basis, this being the most satisfactory method from a comparative point of view, such a basis is not practicable since copra as sold always has a certain moisture content. Further, it will be realised that, other things being equal, from the point of view of oil recovery the lower the moisture content the greater will be the oil content, that is to say, the oil content varies inversely with the moisture content.

The following table illustrates this point. Assuming that the oil content of the copra, calculated on a moisture-free basis, is 66 per cent the calculated percentages of oil in copra

in which the moisture increases from nil to 10 per cent. will be as follows:—

Moisture content per cent.	Oil content per cent.
Nil	66.0
1.0	65.3
2.0	64.7
3.0	64.0
4.0	63.4
5.0	62.7
6.0	62.0
7.0	61.4
8.0	60.7
9.0	60.1
10.0	59.4

The results show that with an increase of one per cent in the moisture content of the copra there is a corresponding decrease of from 0.6-0.7 per cent in the oil content.

Remarks and Conclusions.

The results of the investigation show that the average moisture content of estate copra is approximately 7 per cent. During the period of transit there will be a loss in weight due to further drying dependent for example on the conditions of stowage, time of transit and decrease in the humidity of the atmosphere. Further, the amount of decrease in weight will vary with the moisture content of the copra when despatched.

As far as can be ascertained the average moisture content of copra on arrival in Europe varies from 3 to 4 per cent so that, if the moisture content of the copra as shipped is 7 per cent, the loss in weight during transit will vary from 2 to 3 per cent.

As regards the oil content the results show that apart from seasonal variations the average figure, calculated on a moisture-free basis, is approximately 66 per cent.

It is difficult to suggest a reason for the alleged diminution in oil content, which is probably due to a variety of causes. For example, it is well-known that the oil content

of the brown testa is considerably less than that of the white meat and it is possible that as a result of cultivation under local conditions the thickness of the testa may have increased with a consequent diminution in the oil content of the product. It is intended at an early date to commence a series of experiments to obtain information regarding the proportions of these two constituents.

Summary.

1. The paper deals with an investigation regarding the present oil content of Malayan estate copra.

2. The results show that apart from seasonal variations the average oil content, calculated on a moisture-free basis, lies between 65.5 and 66.0 per cent.

In conclusion the writer wishes to thank both the Managers of the various estates for their kindness in supplying the necessary samples and Mr. Gunn Lay Teik for carrying out the analytical work in connection with the investigation.

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The Cobaltinitrite method for the Estimation of Potash with particular reference to soils.

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In recent years much attention has been called to the estimation of potash by the cobaltinitrite reaction first discovered by W. Fischer in 1848. [1]

Notes on Previous Investigations.

Gilbert [2] in 1898 seems to have been the first to use this method for the estimation of potassium, though he used it solely as a means of separation of the potassium from other metals, subsequently dissolving in hydrochloric acid and reprecipitating as chloroplatinate or perchlorate.

In 1900 Addie and Wood [3] proposed both a gravimetric and volumetric method for the estimation of potash by filtering the cobaltinitrite precipitate through a gooch crucible and weighing as sodium potassium cobaltinitrite or subsequently breaking up the complex with sodium hydroxide and estimating the sodium nitrite formed with potassium permanganate. At the same time they proposed a method for the estimation of potash in soils, by first precipitating the iron and alumina in the soil extract with sodium carbonate filtering off the precipitate, acidifying with acetic acid and subsequent precipitation of the potassium as cobaltinitrite.

This method does not seem to have been taken up generally possibly owing to the fact that the authors indicated the difficulty of washing the precipitate.

In 1909 Mary Cunningham and A. M. Perkin [4] in the course of work on organic cobaltinitrites remarked that the sodium potassium cobaltinitrite precipitation could not be recommended for the estimation of potash owing to the extreme difficulty of washing. "It can easily be washed once by decantation, on filling the vessel with water the precipitate settles very slowly and on the third washing shows a strong tendency to become colloidal. Finally it becomes almost impossible to filter satisfactorily."

Hamberger [5] in 1915 proposed to estimate potassium by measuring the volume of the cobaltinitrite precipitate in a special type of centrifuge tube. A modification of this method has been used by Walker and Glich for the estimation of potash in sugar cane juices.

More recently in 1928 M.M. Leulier, Velluz and Griffon [6] published a summary of previous work, together with researches of their own for the estimation of potash (between 0.1 mg. and 5 mg.) from the biological side. They again proposed the use of the centrifuge for the separation of the cobaltinitrite precipitate, subsequently drying off their precipitate with ether after washing with an alcohol, ether and acetic acid mixture. Finally they added sodium phosphate and estimated the nitrous groups with permanganate and iodine in the cold, thus eliminating losses of permanganate by heating.

Present Investigations.

An examination of the cobaltinitrite method for the estimation of potash was carried out in this department primarily with a view to its application to the estimation of potash in soils, it being recognised that estimation with perchlorate led to very widely varying results, while for routine work estimation as chloroplatinate is far too expensive. For these reasons the Dutch authorities in Java have done little with potash estimations.

What seemed to be required therefore was a rapid method for the estimation of potash which at the same time should shew a good degree of accuracy.

Cobaltinitrite Reagent.

In the work to be described below the sodium cobaltinitrite reagent used was made up according to the formula of Walker and Glich[7] and was found to give very satisfactory results.

(*a*) *Cobalt Acetate Stock Solution.*—125 grams of the pure salt ($Co(CH_3COO)_2 3H_2O$) dissolved in 400 cc. distilled water.

(*b*) *Sodium Nitrite Solution.*—225 grams of the pure salt dissolved in 400 ccs. distilled water. Portions made up when required.

(c) *Sodium Cobaltinitrite Solution for use.*—100 cc. each of (a) and (b) made up to 430 ccs. with distilled water. 12.5 ccs. glacial acetic acid is added and the whole, with occasional stirring, left to stand for 24 hours. It is then filtered and kept in a dark stoppered bottle. It has been found that the reagent keeps fairly well if unexposed to light, but not more than a week's supply should be made at a time.

The Precipitate.

As far as can be ascertained previous workers do not seem to have reported the fact that there two distinct types of sodium potassium cobaltinitrite precipitate of similar composition which can be obtained by different methods of precipitation, though Addie and Wood[3] in their paper mention that potash should be present in a concentration of 0.5 to 1 per cent. in order to obtain a precipitate which settles readily.

It was found that using a decinormal solution of KCl ($K_2O = 0.47\%$) a bright orange yellow precipitate is formed which settles very rapidly. With rather weaker solutions a bright orange precipitate is also formed, precipitation does not occur immediately as with decinormal solutions, but only on standing for some minutes. With still weaker solutions the precipitate is yellow.

With normal solutions of KCl an immediate precipitate is obtained, but in this case it is canary yellow in colour, similar to that obtained with the most dilute solutions. The colour of the precipitate does not depend necessarily on the concentration of the potash but in some cases on the concentration of other salts as well. If solid sodium chloride is placed at the bottom of a tube containing a decinormal solution of potassium chloride (which normally gives an orange precipitate) a canary yellow precipitate is also obtained.

From microscopic examination it would appear that the differences of the precipitates obtained under differing conditions are due to differences in the size of the crystals obtained rather than differences of form. From very dilute solutions of potash the crystals obtained appear to be of a fairly constant size (about 0.004 mms. in diameter) and cubic in form. With decinormal solutions crystals of much the same size and ap-

pearance as those obtained with solutions of the centinormal order seem to be formed first. On standing a minute however they appear to coalesce to form very well defined crystals cubic in shape and about .016 mm. in diameter.

In the case of normal solutions, solutions where the general salt concentration is very great, or in extremely dilute solutions where the canary yellow colour is obtained, the precipitate appears to be colloidal except under very high power, when again the same cubic form can be seen. In this case however the size seems to vary considerably and lies between 0.0015 mm. and 0.0007 mm.

All three precipitates seem stable in form except for the preliminary coalescing with decinormal solutions and after standing for twenty four hours they do not shew any tendancy to change.

It was thought at first that differences in appearance, even in the presence of excess of the reagent might be due to differences in composition, but subsequent examination of the cobalt content disproved this.

Not only is there a considerable difference in the appearance of the precipitates but there is a very considerable difference in the volume of the precipitate obtained (though not in weight) which seems to detract from the accuracy of centrifugal methods based on the estimation of the volume of precipitate obtained unless special precautions are taken that the concentration of other salts in the standard potash solution shall be the same as in the solution under examination. It has been found that the relative volumes of precipitate using decinormal and normal solutions of potassium chloride are between 1 to 3 and 1 to 4.

The precipitates shew very great differences in their rate of centrifuging, the orange precipitate obtained from decinormal solutions settles in about a minute at speeds of about 800 revolutions per minute even after several washings. The yellow precipitate settles fairly rapidly at first (as was pointed out by Mary Cunningham and Mollwo Perkin) but after washing there is an increasing tendancy to remain in suspension and as much as fifteen minutes are required to settle it. As would be expected with precipitates obtained from very dilute solutions, where the crystal size lies between that ob-

tained from decinormal and normal solutions the centrifuging time lies mid way between the two.

From work carried out in this laboratory it appeared that centrifuging was certainly the surest and most convenient method for the separation of the potash precipitate from the mother liquors.

The centrifuge tubes in use were made of ordinary pyrex test tubes* drawn out at the bottom to pear shape. This shape was found to help separation considerably, forming a pocket into which the precipitate could sink and this allowed the supernatant liquor to be poured off completely.

The potash solution to be examined is poured into the tube first and the cobaltinitrite reagent added in considerable excess. The tubes are then allowed to stand for twenty minutes as it was found that although the precipitation is immediate for amounts of potash of the order of 2 milligrams and over with considerably smaller amounts the precipitate only comes down on standing. The tubes are then lightly corked and centrifuged until separation is complete and the precipitate well bedded down into the pear shaped depression. The mother liquor is completely decanted, replaced with water, the tubes thoroughly shaken up and recentrifuged. The washing is repeated a second time, the wash water again decanted after centrifuging and the precipitate examined by the methods given below.

It has been found that by centrifuging in this manner, getting the precipitate well bedded down each time of centrifuging that a very complete separation is obtained, no subsequent washing, drying or filtration being necessary Eight separations can be made at once and the whole operation completed in 15 minutes.

In applying the cobaltinitrite methods to soils in Malaya where large numbers of soils have to be operated on by semi skilled assistants it appeared essential that the final estimation of the potash should be made as simple as possible. It was

* As no centrifuge capable of taking test tubes was available in the laboratory and none such could be found in any of the scientific catalogues, one was built up of "Meccano" parts (obtainable from Meccano Ltd., Binns Road, Liverpool) and driven from a small electric motor by a belt. The cups to carry the tubes were made of galvanised sheet and were coupled to the centrifuge by Meccano chain. The apparatus had been found very satisfactory and of great general utility for the rapid settling of precipitates in test tubes.

decided therefore to try whether the complex precipitate could be broken up by decinormal sulphuric acid and the excess of acid which had not combined with the metallic radicles titrated back to neutrality with decinormal soda used Brom-Thymol-Blue as indicator.

From the results of over sixty estimations by this method it was found that 1 cc. N/10 KCl was equivalent to 1.66 ccs. N/10 H_2SO_4 which would appear to indicate that a precipitate $K_3Co\ (NO_2)_6$ had been obtained. Subsequent estimations of the cobalt and the presence of sodium shewed however that the normal precipitate $K_2NaCo\ (NO_2)_6$ had been obtained. The difference therefore between a value of 1 cc. N/10 KCl = 1.66 CC N/10 H_2SO_4, and a value of 1 cc. N/10 KCl = 2.5 cc. N/10 H_2SO_4 which should be obtained with $K_2NaCo\ (NO_2)_6$ must be due to the presence of an acid set free during the reaction.

Examination shewed the presence of nitric acid in the sulphuric acid solution in the equivalent amount.

The reaction was also carried out in an atmosphere of CO_2 and the gas evolved was collected over caustic soda. This was found to consist chiefly of nitric oxide. Collection over paraffin shewed the fact that either a certain amount of nitrogen peroxide is also evolved, or that a small amount of oxygen is evolved and immediately combines with some of the nitric oxide.

The method compares well with the permanganate one for amounts of potash up to 23 milligrams but for potash present in larger amounts it is better to take an aliquot for its estimation by either method.

As 1 cc. of N/10 K_2O is equivalent to 1.66 cc. N/10 H_2SO_4 or to 5.5 cc. N/10 $KMnO_4$ it is of course obvious that the latter reagent gives a greater degree of delicacy and should be used when the amount of potash present is of the order of 1 mg.

Application to Soils.

In applying the method to the estimation of potash in soil it was found that the proposal of Addie and Wood worked well even when, as happens in Malayan soils the HCl extract may contain as much as 20% of iron and alumina and as little as 0.01 per cent. of potash.

The method they suggested consists in the precipitation of iron and alumina with sodium carbonate (a salt which is generally free from potash) with subsequent boiling and filtration of the precipitated carbonates, acidification with acetic acid and evaporation to a bulk of about 10 cc. (or such bulk as gives a K_2O concentration of about 0.5 to 1%).

This has been modified to some extent in this laboratory. An aliquot of the hydrochloric acid extract equivalent to 10 gms. of soil is neutralised as accurately as possible with sodium carbonate using an external indicator (the thickening of the precipitate near the neutral point with the large bulk of iron and aluminium carbonates obtained with local soils, is in itself sufficient indication.) The solution and precipitate is then made to some convenient volume and the whole centrifuged until separation of the iron and aluminium is complete. This centrifuging where the amount of precipitate is large saves a very considerable time in filtering and does not interfere with the accuracy of the estimation. After centrifuging, the whole or an aliquot of the clear liquor (the volume of which is measured) is evaporated as near to dryness as possible in a glass basin on the water bath, acidified with acetic acid and then treated with cobaltinitrite reagent in the ordinary way. It should be noted that with soils high in iron and alumina, the centrifuged liquor from which, will be high in sodium chloride may give the yellow precipitate instead of the larger crystalled orange one, and in consequence take longer to centrifuge.

As iron and alumina carbonate are soluble to some extent a small precipitation of these substances occurs when the centrifugal liquor is evaporated to small bulk. These however redissolve on addition of the acetic acid.

Addie and Wood recommend the addition of acetic acid before evaporation; there is a subsequent precipitation of basic acetates which interfere to some extent with the potash precipitation. This is avoided by addition of acetic acid subsequently, the iron and alumina then being held on solution as the normal acetates.

In order to ascertain whether potash was occluded in the iron and aluminium precipitates a number of artificial soil solutions were made up containing over 20% of iron and aluminium chlorides and known amounts of potash added. The results obtained are given in the table.

With the idea of applying it to soils to a solution containing varying amounts of citric acid varying amounts of a N/10 solution of potassium chloride were added. The citric acid was then neutralised with sodium carbonate and the solutions evaporated to small bulk. Cobaltinitrite reagent was added and the potash precipitate centrifuged out in the ordinary way. The figures obtained are given in the attached table.

Summary.

The different precipitates obtained under different conditions by the addition of sodium cobaltinitrite solution to a solution of a potassium salt are described.

2. The use of a centrifugal method of separation of the potassium sodium cobaltinitrite precipitate recommended by other writers is strongly advocated.

3. The absence of any necessity for further treatment of the cobaltinitrite precipitate after centrifuging previous to its estimation is indicated.

4. For rapid work where the highest degree of accuracy is not required and the amount of potash lies between 2 and 33 milligrams the solution of the precipitate in sulphuric acid and subsequent titration with soda using brom-thymol-blue is suggested. With smaller amounts of potash or where a high degree of accuracy is required, the solution of the precipitate in acid potassium permanganate, subsequent addition of excess of N/10 oxalic acid and back titration with potassium permanganate is preferable.

5. A modified application of the cobaltinitrite method to potash in soils recommended by Addie and Wood is described.

6. The use of a larger centrifuge for the rapid separation of the iron and aluminium precipitate is advocated.

7. It is shewn that the potash contained in a citric acid solution can be estimated by this method and it is suggested that this might be applied to the estimation of "available potash" in soils.

BIBLIOGRAPHY.

1. FISHER, W. Ann. Ph. Pogg. 1848. **14.** 115.

2. GILBERT, K. Die Bestimmung des Kaluim nach quantitative Abscheidung desselben als Natrium Kobaltinitrit. Inaugural Address Tuubingen 1898.

3. ADDIE, H. A. and Wood, T. B. Volumetric Estimation of Potash by cobaltinitrite of sodium. Journ. of Chem. Soc. 1900. **17.** 1076.

4. CUNNINGHAM, M. and PERKIN, F. M. Studies on the cobaltinitrites. Journal of Chem. Soc. 1909. **95.** 1562.

5. HAMBERGER, H. S. Eine einfache Methode zur quantitativen Bestimmung sehr geringer Kaluimmengen. Biochem. Ztschr. 1915. **71.** 415.

6. LEULIER A, VELLUZ and GRIFFON, H. Sur le microdosage du potassium sour forme de cobaltinitrite. Bull. Soc. Chim. Biol. 1928. **x.** 891.

7. WALKER, H. and GLICH, G. B. Determination of Potash in cane juices as an indication of the fertiliser requirements of soil. Inter. Sugar J. 1923. **25.** 478.

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TABLE.

Using solutions of Potassium Chloride, Potassium Sulphate or Potash Alum.

Potash Alone		In Excess NaCl.		In excess $MgCl_2$ and $CaCl_2$		In large Excess $FeCl_3$ (1.4 gms) and $AlCl_3$ (6 gms) with precipitation of $F_2(CO_3)_3$ and $Al_2(CO_3)_3$		In small amounts of $FeCl_3$ (0.1 gms) and $Al_2(SO_4)_3$ (0.5 gms) without precipitation of $Fe_2(CO_3)_3$ and $Al_2(CO_3)_3$		In excess Citric acid after neutralising with Na_2CO_3 (Potash free)			Solution of ppte. in acid $KMnO_4$ etc. 1 cc. N/10 K_2O= 5.5 cc. N/10 KMnO or 1 cc. N/10 $KMnO_4$=0.855 mg K_2O	
K_2O added in mg.	K_2O found in mg.	K_2O added in mg.	K_2O found in mg.	K_2O added in mg.	K_2O found in mg.	K_2O added in mg.	K_2O found in mg.	K_2O added in mg.	K_2O found in mg.	Citric in gms	K_2O added in mg.	K_2O found in mg.	K_2O added in mg.	K_2O found in mg.
4.7	4.65	4.7	4.7	4.7	5.0	47.0	49.0	9.3	9.3	5	23.5	23.0	23.5	23.9
4.7	4.65	4.7	4.5	9.4	9.6	42.3	41.0	9.3	8.8	5	23.5	23.5	23.5	23.2
9.4	9.0	9.4	9.3	9.4	9.3	37.6	34.0	9.3	8.6	5	14.1	14.1	9.4	10.2
9.4	9.3	4.7	4.1	14.1	14.6	32.9	32.7	9.3	7.9	5	14.1	13.3	9.4	9.8
23.5	23.2	9.4	9.1	18.8	17.7	28.2	29.0	9.3	9.3	2	14.1	14.1	23.5	23.8
14.1	14.5	9.4	9.2	18.8	18.8	9.4	9.8	9.3	9.3	2	14.1	13.5	9.4	10.3
14.1	13.9	23.5	23.5	23.5	24.0	4.7	4.3	9.3	8.8	2	4.7	4.2	18.8	19.4
18.8	18.8	28.2	26.2	4.7	4.7	18.8	18.5	9.3	9.0	2	4.7	4.2	32.9	32.5
9.4	9.9	18.8	19.0			18.8	18.2	9.3	9.3					
4.7	4.9	14.1	14.3			23.5	23.0							
9.4	8.9					32.9	33.6							
18.8	18.6					32.9	32.9							
23.5	23.6													
28.2	28.2													
32.9	31.8													
37.6	36.8													
14.1	14.4													
18.8	18.8													
9.4	9.0													
4.7	4.1													
23.6	23.5													
9.4	9.3													
etc.	etc.													

Solution of $K_2NaCo(NO_2)_6$ in decinormal H_2SO_4
Mean of 60 Estimations 1 cc. N/10 K_2O=1.66 ccs.
N/10 H_2SO_4 or 1 cc. N/10 H_2SO_4=2·82 mg. K_2O.